

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:) Art Unit: 1793
Gunter Kaupp)
Examiner: Shuagngyi Aub Ali
Appln. No.: 10/525,395) Washington, D.C.
I.A. Filed: 08/07/2003) Confirmation No. 7134
371 (C): 02/22/2005)
For: PIGMENT AND PIGMENTED) ATTY.'S DOCKET: KAUPP=1
COSMETIC PREPARATION...

DECLARATION UNDER 37 CFR 1.132

I, Dr. Hans-Jörg Kremitzl, hereby solemnly declare
as follows.

1. An abbreviated form of my *Curriculum Vitae*
follows:

- 1985 - 1992: Study of Chemistry at Friedrich
Friedrich-Alexander University Erlangen-Nuremberg;
- 1992 - 1995: PhD in Inorganic Chemistry at
Friedrich-Alexander University Erlangen-Nuremberg;
- 1995 - 1996: further education in Quality
Management (DIN EN ISO 9001);
- since 1997: employed at ECKART GmbH (Group
Leader in R&D, responsible inter alia for the coating of
effect pigments with inorganic (silica) layers, since
2007 Head of Quality Coordination - Process Quality).

2. I am one of the co-inventors of the invention of the above-identified application, and am very familiar with its contents, and with the prior art, including the prior art applied against our claims in the Final rejection of September 25, 2008; and I am also familiar with the commentary of the U.S. examiner in the Advisory Action of February 2, 2009.

3. In USP 5,607,504, Schmid et al describe aluminum pigments containing Silica in amounts of 25.0% to 52.4% which were deposited with sol-gel or CVD methods and isolated for further use. The examiner states that these intermediate products could theoretically be used in cosmetic formulations, similarly to the products described in our U.S. Patent Application No. 10/525,395, and that (bottom of page 3 of the final rejection) "it would be expected that the pigment disclosed by the references has the same or similar properties absent any evidence to the contrary."

4. To show the important differences between our invention and Schmid et al, contrary to the position of the examiner, certain experiments were conducted under my supervision as follow.

Experimental Findings:

5. Advantages of the pigments according to our No. 10/525,395 are the excellent optical properties with respect

to brilliance, hiding power, and brightness together with a good chemical resistance, e.g. against sweat.

6. To emphasize the differences in the optical properties and stability, aluminum pigments were manufactured according to Example 1a of Schmid USP 5,607,504. The reaction was conducted for different times between 2 - 5 hours and the samples contained different amounts of silica, accordingly. The initial aluminium pigment had a specific surface (BET) of about $0.8 \text{ m}^2/\text{g}$. These samples were compared to pigments manufactured according to our present invention with relatively low silica content (Visionaire Bright Silver Sea, based on an aluminum pigment with BET = $1.5 \text{ m}^2/\text{g}$).

7. The pigment disclosed in example 4 of Schmid et al differs in several aspects from the pigments of our invention. First of all, very coarse aluminium flakes ($D_{50} = 60 \mu\text{m}$) are used in Schmid et al. The size of the pigments of our invention is restricted to $D_{95} < 45 \mu\text{m}$. Such coarse pigments will lead to a completely different optical impression in cosmetic formulations. Such coarse pigments lead to a sparkle effect whereas our pigments lead to a smooth silver shiny impression. Such coarse pigments also have greater tendency to irritate the skin.

8. Secondly, the method of silica deposition described in example 4 is not a usual sol-gel process as the

particles are not suspended in an organic solvent. Here a CVD (chemical vapor deposition) process is described.

9. As a third point, silica is deposited in higher thickness than the claimed 20 - 50 nm.

10. The thickness of the silica layer in Schmid et al can be easily calculated by the following formula:

$$d_{\text{SiO}_2} = V_{\text{SiO}_2} / A_{\text{Al}} = m_{\text{SiO}_2} / (\rho_{\text{SiO}_2} \times m_{\text{Al}} \times \text{BET}_{\text{Al}})$$

1 g of silica coated pigment contains 0.75 g of aluminium and 0.25 g of silica. Using the density of bulk amorphous silica $\rho_{\text{SiO}_2} = 2.4 \text{ g/cm}^3$ and the given specific surface of the initial aluminium pigment:

$$d_{\text{SiO}_2} = 0.25 \text{ g} / (2.4 \text{ g/cm}^3 \times 1.5 \text{ g/m}^2 \times 0.75 \text{ g}) = 0.926 \text{ cm}^3/\text{m}^2 = 93 \text{ nm.}$$

Silica thicknesses in these high ranges have an impact on the color of the coated pigment, i.e. lead to greenish to reddish tinge as observed by the inventor. This is in contrast to the pigments according to our 10/525,395 where the original color of the pigment is not impaired.

11. Both pigments grades were then analyzed with respect to their chemical composition, particle size, optical properties, (hiding power), and chemical stability:

- Chemical Composition: The silica content was determined via X-Ray fluorescent analysis, due to the fact that it is difficult to gravimetrically determine

the residue of ignition after the dissolution of the metallic core due to the chemical resistance of the silica layer.

- Optical Properties: The pigment was dispersed in a Nitro cellulose lacquer (5%) and applied on a black/white opacity display chart using an automatic drawdown applicator equipped with a spiral wire bar. The finished drawdown was measured with a photospectrometer. The difference of the L* values (lightness) between the black and the white section of the chart correlates with the hiding power of the pigment. The smaller the ΔL^* values the better is the hiding power of the pigment.

- The particle size was determined using Laser diffraction methods (Cilas 1064) and expressed as the D50 value, e.g. the value where 50% of the volume weighted particle size distribution is below the respective value.

- Chemical stability was determined by dispersing the pigments in an aqueous formulation and monitoring the evaluation of hydrogen at 40°C. Higher values indicate less stability.

Test results:

| ID # | L value white | L value black | ΔL | Silica content | Calculated Silica | Particle Size (D50, | Gassing |
|------|------------------|------------------|------------|-------------------|----------------------|------------------------|---------|
|------|------------------|------------------|------------|-------------------|----------------------|------------------------|---------|

| | section | section | thickness | | | μm) |
|----------------------|---------------|---------|-----------|------|------|---------------|
| | | | [nm] | | | |
| NBD 00401-91-2 | ¹⁾ | 87.5 | 70.7 | 16.8 | 9 % | 51 61.5 - |
| NBD 00401-91-3 | ¹⁾ | 88.2 | 63.3 | 24.9 | 18 % | 114 63.2 - |
| NBD 00401-91-4 | ¹⁾ | 89.3 | 56.3 | 32.9 | 24 % | 165 66.0 - |
| NBD 00401-91-5 | ¹⁾ | 89.4 | 54.1 | 35.3 | 27 % | 193 71.5 8 ml |
| Visionaire Bright | | | | | | |
| Silver Sea (08G0001) | ²⁾ | 89.0 | 88.7 | 0.3 | 9 % | 27 22.8 3 ml |

¹⁾ Pigment according to USP 5,607,504. The last digit indicates the time of the sol gel reaction.

²⁾ Pigment according to current invention (USPA 10/525,395)

12. The table above shows clearly that the pigment according to the current invention has a significantly improved hiding power in comparison to a pigment according to the invention of Schmid et al. This higher level of hiding power leads to a significantly enhanced tinctorial power in any cosmetic formulation.

13. The better hiding power has two reasons: firstly the coarser aluminium pigment has naturally a lower hiding power, as coarser pigments are also thicker. The second reason is the formation of agglomerates in the samples NBD 00401-91 of Schmid et al which drastically reduce the hiding power. Agglomerates could be well seen visually on the draw downs by the naked eye as well as under an optical microscope. The larger the content of SiO₂ the higher was the tendency of agglomerates. Hence the pigment manufactured

according to our invention has a much lower tendency of agglomeration during the manufacturing process.

14. The formation of agglomerates is also substantiated by the values from the particle size measurement, which increase with an increased amount of silica.

15. Additionally the chemical stability of said coated pigments is significantly higher despite the fact that the silica content of these pigments is reduced in comparison to the pigments according to Schmid USP 5,607,504, and despite the fact that the hiding power and hence the theoretically reactive surface of said pigments is much higher.

16. I hereby conclude based on the above reported experiments that our pigments are very different from that of Schmid, and do not have the same or similar properties.

I hereby further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

By

Dr. Hans-J. Kremitz
Dr. Hans-Jörg Kremitz

Date: May 15th, 2009